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Indian Standard

TEXTILES — ESTIMATION OF MOISTURE, TOTAL SIZE OR FINISH, ASH AND FATTY MATTER IN GREY AND FINISHED COTTON TEXTILE MATERIALS

(Third Revision)

भारतीय मानक

धूधर और तैयार सूती वस्त्रादि सामग्रियों में नमी, कुल माड़ी अथवा फिनिश, राख और वसीय पदार्थ का मूल्यांकन

(तीसरा पुनरीक्षण)

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NEW DELHI 110002

October 1989 **Price Group 3**

Chemical Methods of Test Sectional Committee, TDC 5

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards on 15 May 1989, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

This standard, first published in 1956, was revised in 1957 and 1973. The present revision has been taken up to make it up to date on the basis of experience gained during its use. In this revision, the procedure for estimation of starch, fatty matter and ash has been modified.

The size used for spun textile yarns may consist of an adhesive, a lubricant, an antiseptic, or hygroscopic and weighting materials. The adhesive is generally starch or soluble starch, but certain other carbohydrates, for example, locust-bean gum, may be used alone or in mixtures with starch. The filling applied to fabric in finishing may contain starch or dextrins with or without lubricant, and sometimes substantial proportion of weighting materials, such as, china clay. The procedure described for estimation of starch is for use where test specimens of about 2 g are available; weighting materials, if present, are mainly removed along with the starch; the residual filling may be determined by ashing the desized specimen and, provided that the amount of residual mineral matter is small, changes in its mass produced during ashing may be neglected. Unsized grey cotton loose mass in desizing process and a correction factor for the loss, has to be applied to the mass of the residual. The correction can be determined if a specimen of the corresponding unsized yarn is available. In the absence of such a specimen, the values for the correction factor are assumed.

The oils, fats and waxes present in cotton may be derived either from the cotton itself, or from sizing and finishing compositions added during manufacture. With some exceptions, these materials are removed by extraction with suitable solvent. Dichloromethane (methylene chloride) has been found as suitable solvent for grey, scoured and bleached cotton if the extraction time does not exceed six hours, but it is unsuitable for dyed or printed materials containing vat or azoic dyes or colourants for which the use of light petroleum (boiling range 40 to 60°C) is recommended. The amount of natural fat or wax extracted from cotton materials by light petroleum is smaller than that extracted by dichloromethane. The method prescribed does not purport to remove natural fats and waxes completely. It gives a result that may be low by about 0·2 g per 100 g of cotton or raw cotton, and low by less than this amount on partly purified cotton but which is nevertheless, reproducible and serves to characterize the cotton sufficiently for practical purposes.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding of numerical values (*revised*)'.

Indian Standard

TEXTILES — ESTIMATION OF MOISTURE, TOTAL SIZE OR FINISH, ASH AND FATTY MATTER IN GREY AND FINISHED COTTON TEXTILE MATERIALS

(Third Revision)

1 SCOPE

1.1 This standard prescribes methods for estimation of (a) moisture, (b) total size or finish, (c) ash and (d) fatty matter in grey and finished cotton textile materials.

1.2 The method prescribed for estimation of starch is not applicable in cases where the starch has been insolubilized as in formaldehyde-containing finishes and is not intended for use in determining small amounts of residual starch and fatty materials. The method prescribed for determination of fatty matter is not applicable to textile materials containing oxidized unsaturated oils, waterproofing or softening preparations derived from cetylpyridinium halides, rot-proofing agents, synthetic resins and other substances that would interfere.

2 SAMPLING

2.1 Samples from the lot for testing shall be selected so as to be representative of the lot.

2.2 Samples drawn in accordance with the procedure laid down in the material specification or as agreed to between the buyer and the seller shall be held to be representative of the lot.

3 QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4 ESTIMATION OF MOISTURE

4.1 Apparatus

4.1 1 *Drying Oven*, capable of maintaining a temperature of 105 ± 3°C

4.1.2 *Weighing Balance*

4.2 Procedure

From the sample under test (*see* **2**) draw at least 2 test specimens each weighing approximately 3 g.

4.2.1 Take one test specimen drawn as in **4.2** and weigh it accurately in a clean and dry tared weighing bottle. Place the weighing bottle containing the test specimen in the drying oven and dry the specimen at 105 ± 3°C to constant mass (*see* Note) and determine the oven-dry mass of the test specimen.

NOTE — The mass may usually be regarded as constant if the loss between two successive weighing, taken at an interval of 20 minutes, does not exceed by 0·1 percent of the first of the two values.

4.2.2 Similarly test the other test specimen(s).

4.3 Calculations

Calculate the percentage of moisture content in the test specimen by the following formula:

$$\text{Moisture content, percent} = \frac{(a - b)}{a} \times 100$$

where

a = original mass in g of the test specimen, and

b = oven-dry mass in g of the test specimen.

4 3.1 Determine the mean of all values, obtained in **4.3** and express it as moisture content of the material in the lot.

4.3.2 Corrected invoice mass of cotton textile materials may be calculated by the following formula:

$$L_1 = \frac{L (b \times 1{\cdot}085)}{a}$$

where

L_1 = corrected invoice mass in g of the lot,

L = original mass in g of the lot,

b = oven-dry mass in g of the test specimen, and

a = original mass in g of the test specimen.

5 ESTIMATION OF TOTAL SIZE OR FINISH

5.1 Apparatus

5.1.1 *Soxhlet Extractor*

5.1.2 *Drying Oven*, capable of maintaining a temperature of 105 ± 3°C.

5.1.3 *Sintered Disc Filter Crucibles*, of at least 60 ml capacity, fitted with ground glass stopper or other suitable cover.

5.1.4 *Suitable Ignition Crucibles and Covers*

5.1.5 *Muffle Furnace*, or other equipment for heating crucibles to a temperature of about 750°C.

5.2 Reagents

5.2.1 *Dichloromethane* (*Methylene Chloride*), redistilled.

5.2.2 *Diastase or other Suitable Desizing Enzyme*

5.2.3 *Sodium Chloride*

5.2.4 *Soap-Soda Solution*

It shall contain 20 g of soap and 10 g of anhydrous sodium carbonate per litre. The soap shall contain not more than 5 percent moisture and shall comply with the following composition on dry mass basis:

Free alkali (as Na_2Co_3), *Max*	0·3 percent
Free alkali (as NaOH), *Max*	0·1 percent
Combined fatty acids (as sodium salt), *Min*	85 percent
Titre of mixed fatty acids prepared from the soap, *Max*	30°C
Iodine value of fatty acids, *Max*	50

5.3 Procedure

From the sample under test (*see* **2**), draw at least 2 test specimens each weighing approximately 2 g.

5.3.1 If the sample under test is yarn, cut each test specimen, into pieces of 15 cm long, form into separate bundles and tie each bundle loosely round the middle.

5.3.2 If the sample under test is fabric, trim each test specimen parallel to the directions of warp and weft and pull out 5 threads all round to form a fringe.

5.3.3 Determine the moisture content of the sample by the method prescribed in **4**, using separate test specimens. Weigh accurately one test specimen in a tared crucible drawn as in **5.3** and calculate its oven-dry mass. Place the crucible with specimen in a Soxhlet apparatus, with its rim above the level of the siphon and extract it in a Soxhlet extractor for one hour with dichloromethane adjusting the rate of boiling to at least six hot extractions per hour.

5.3.4 Remove the crucible from the extractor and allow the solvent to evaporate. Attach the crucible to a Buchner flask and wash the extracted specimen by drawing about 100 ml of water through it. Drain and remove the crucible and transfer the contents to a flask of 150 ml capacity.

5.3.5 Treat the extracted specimen in the manner prescribed in **5.3.5.1**, **5.3.5.2** or **5.3.5.3** depending on the type of the ingredients used in the size or finish.

5.3.5.1 In case it is known that the material is sized or finished with a mixing containing starch but not tamarind kernel powder, add to the flask 50 ml of bacterial diastase solution containing 5 g/l of active diastase, 10 g/l sodium chloride and 0·1 percent (*m*/*m*) of a non-ionic wetting agent at 70°C and at a *p*H of 6·5 to 7·5 (*see* Note). Maintain this temperature for 30 min and shake the contents of the flask by any convenient means continuously or at intervals of 5 min during this time. Decant the solution through the filter crucible, add a fresh portion of bacterial diastase solution at 70°C and at a *p*H of **6.5** to **7.5** to the flask and repeat the desizing treatment. Take precaution to ensure that the temperature in any part of the solution does not exceed 70°C or the limiting temperature specified by the manufacturer of enzyme. After decanting the second portion of the solution, add water to the flask and boil for 5 min. Finally transfer the desized material to the crucible, drain and wash with three successive 100 ml portions of hot water while stirring the contents with a glass rod, and drain with suction after each wash. If the washings are opalescent with mineral filling, continue washing until they are clear.

NOTE — The temperature and *p*H given for the desizing solution are the optimum for bacterial diastase. If any other type of desizing enzyme is used, then the temperature and *p*H should be modified to that recommended by the supplier. As many enzymatic desizing agents slowly deteriorate in storage, care should be taken to see that the sample of desizing agent, at the time of test, has still satisfactory desizing efficiency.

5.3.5.2 In case it is known that the material is sized or finished with a mixing containing tamarind kernel powder but not starch, boil the specimen in the soap-soda solution (weighing 20 time the mass of the specimen) for 45 minutes. Wash it thoroughly first in hot water and then in cold water.

5.3.5.3 In case it is known that the material is sized or finished with a mixing containing both starch and tamarind kernel powder or in case the ingredients used in the size or finish are not known desize the specimen as prescribed in **5.3.5.1** and treat it further as prescribed in **5.3.5.2.**

5.3.6 Dry the specimen treated as above (*see* **5.3.3** to **5.3.5**) in the drying-oven at 105 ± 3°C to constant mass (*see* Note under **4.2.1**) accurately.

5.3.7 *Residual Mineral Filling*

If the sample originally contained a substantial amount of insoluble mineral filling, for example, china clay, transfer the desized specimen to an ignition crucible, previously ignited with its lid at 750°C (*see* Note 3 under **6.2.1**), cooled and its mass determined. Place the open crucible at the front of the muffle furnace and burn off the fibre while manipulating the crucible lid to prevent the fibre from inflaming. Move the crucible to the interior of the furnace, replace the lid, and continue the combustion at 750°C until no carbonaceous residue remains. Cool and determine the mass of the crucible and its content and calculate the mass of ash. Correct the mass of the desized specimen by subtracting the mass of the ash.

5.3.8 Similarly repeat the test with other test specimens.

5.4 Calculations

Calculate the percentage of total dry size or finish expressed on the oven-dry mass of the unsized material by the following formula:

$$X = \frac{a - kb}{kb} \times 100$$

where

X = total size or finish, percent by mass;

a = oven-dry mass (calculated) in g of the test specimen containing the size or finish;

$K = \frac{c}{d}$, that is, the correction factor determined experimentally (on unsized or blanks) for adjusting the value of b, due to loss of natural constituents sustained by the test specimen during treatment as prescribed in **5.3.3** and **5.3.4;**

b = oven-dry mass in g of the test specimen determined as in **5.3.6** or **5.3.7,** if necessary;

c = oven-dry mass in g of unsized or unfinished blanks; and

d = oven-dry mass in g of blank after treatment as prescribed in **5.3.3** to **5.3.7.**

NOTE — Normally k cannot be determined experimentally as a comparable unsized or unfinished control blank is usually not available, particularly so in the case of fabric. In the absence of a control blank, k shall be taken as equal to 1·05 in the case of grey yarn and fabric; and equal to 1·00 in the case of scoured bleached, dyed, mercerized and printed material. The prescribed values of k for unsized grey cotton material may vary from actual value, particularly if Indian cottons are involved. Therefore, some slight inaccuracy may be expected when the prescribed values are used.

5.4.1 Determine the mean of all values as obtained in **5.4.**

5.4.2 Total size or finish of cotton textile material expressed as a percentage of:

a) original mass of the material,

b) conditioned mass of the material, and

c) corrected invoice mass of the material, may be calculated by the following formula:

$$X_1 = \frac{a - k\,b}{a_1} \times 100$$

$$X_2 = \frac{a - k\,b}{a_2} \times 100$$

$$X_3 = \frac{a - k\,b}{k\,b \times 1{\cdot}085} \times 100$$

where

X_1 = total size or finish as a percentage by mass of the original mass of the material,

X_2 = total size or finish as a percentage by mass of the conditioned mass of the material,

X_3 = total size or finish as a percentage by mass of the corrected invoice mass of the material,

a = oven-dry mass (calculated) of the test specimen containing the size or finish,

k = correction factor for b (*see* **5.4**),

b = oven-dry mass of the test specimen determined as in **5.3.5,**

a_1 = original mass in g of the test specimen, and

a_2 = conditioned mass in g of the test specimen.

6 ESTIMATION OF ASH

6.1 Apparatus

6.1.1 *Silica or Porcelain Crucible*, of about 40 ml capacity.

6.1.2 *Muffle Furnace*, capable of being heated to 750°C.

6.1.3 *Drying Oven*, controlled at 105 ± 3°C.

6.1.4 *Weighing Bottle*, of suitable size and shape to contain the crucible.

6.1.5 *Desiccator (Vacuum Type)*, containing anhydrous calcium chloride.

6.1.6 *Pastille Press*, one, suitable for making pastilles of 15 mm diameter.

6.2 Procedure

6.2.1 Compress a suitable mass of the material (*see* Note 1) into pellets from 0·5 to 1·0 g each and heat in an open, tared, weighing bottle at 105 ± 3°C overnight (*see* Note 2). Place the stopper in the bottle and after cooling in a desiccator, determine the mass of the bottle and its contents. Transfer the pellets to the crucible which has previously been ignited, cooled in desiccator and its mass accurately determined in the weighing bottle. Burn the pellets cautiously at the front of the open furnace, taking care to avoid draughts and when the material has reached the glowing stage, move the crucible towards the rear of the furnace, close the front door, and complete ashing at about 750°C (*see* Note 3). Transfer the dish after it has been allowed to cool in the desiccator, to the weighing bottle and finally determine its mass (*see* Note 3).

NOTES

1 The mass of material used is governed by the anticipated ash, for example, for grey cottons a 5 g specimen is generally used; for a well-scoured or bleached cotton material, a 10 g specimen is needed to give an amount of ash that can be determined with reasonable accuracy.

2 For many purposes, the initial drying of the specimen may be neglected and an average figure assumed for the moisture content of the fibre.

3 Certain metals can be lost under these conditions of heating. Mercury is lost readily and zinc is lost above 450°C. Temperatures in excess of general ashing temperature may occur locally in large specimens and the general ashing temperature in such cases may have to be considerably reduced to avoid loss of certain metals Lead is also lost under the specified conditions of heating unless sulphate ion is present.

4 Considerable loss in ash may occur due to draught. It is particularly necessary to exercise caution in opening the desiccator in which the ash has been cooled since a partial vacuum is created during cooling. The safest procedure is to press a piece of filter paper against the outlet whilst cautiously opening the tap.

5 Silica and porcelain crucibles become etched during heating and cooling cycles over a prolonged period and it is recommended that crucibles should be renewed fairly frequently.

6.2.2 Similarly repeat the test with at least one more test specimen.

6.3 Calculation

Calculate the percentage of ash by the following formula:

$$\text{Ash content, percent} = \frac{a}{b} \times 100$$

where

a = mass in g of the residue (ash), and

b = oven-dry mass (calculated) in g of the test specimen.

6.3.1 Determine the mean of all values as obtained in **6.3**.

7 ESTIMATION OF FATTY MATTER

7.1 Apparatus

7.1.1 *Soxhlet Extractors*, of capacity 200 to 250 ml.

7.1.2 *Flat-Bottomed Extraction Flasks*, of capacity 250 to 300 ml.

7.1.3 *Double-Surface Reflux Condensors*

7.1.4 *Suitable Solvent Removal Apparatus*, all glass wire shall be fitted with interchangeable ground glass joints.

7.1.5 *Drying Oven*, controlled at 105 ± 3°C.

7.1.6 *Thimbles*, previously extracted with the solvent.

7.2 Reagents

7.2.1 *Dichloromethane (Methylene Chloride)*, redistilled.

7.2.2 *Light Petroleum*, redistilled, boiling range 40 to 60°C.

7.3 Procedure

7.3.1 From the sample under test, draw at least two test specimens each weighing about 5 g.

7.3.2 Take one test specimen drawn as in **7.3.1** and weigh it accurately in a clean dry, tared weighing bottle. Place the weighing bottle containing the test specimen in the drying-oven maintained at 105 ± 3°C and dry the specimen to constant mass (*see* Note under **4.2.1**) and note the mass to an accuracy of 0·05 g.

7.3.3 If the material contains size or finish based on starch in which oil, fat or wax has been incorporated, treat the specimen in 0·4 N hydrochloric acid for 30 minutes under reflux at the boil, rinse thoroughly in water and dry at a temperature not exceeding 55°C. Place the specimen in a thimble in a Soxhlet extractor. Dry an extraction-flask at 95 to 100°C, cool in a desiccator and determine its mass. Assemble the apparatus and pour the solvent (*see* Note) slowly through the condenser until the siphon operates. Add a further 10 to 20 ml of solvent. Heat the flask on a water-bath or electric hot plate so that the solvent siphons at least six times per hour for three hours, keeping the volume of solvent fairly constant by adding enough of it to make up for any loss due to evaporation

NOTE — The choice of solvent should be as agreed to between the buyer and the seller.

7.3.4 Disconnect the apparatus and remove the bulk of the solvent by means of the removal apparatus. Evaporate the extract carefully to dryness in the flask on a steam bath, removing the last traces of solvent by means of a jet of clean air. Further dry the contents in the drying oven at 105 ± 3°C to constant mass (*see* Note under **4.2.1**), cool in a desiccator and determine the mass of the specimen to an accuracy of 0·05 g.

7.3.5 Similarly repeat the test with the remaining test specimen(s).

7.4 Calculation

Calculate the percentage of fatty matter by the following formula:

$$\text{Fatty matter, percent, on dry mass basis} = \frac{b}{a} \times 100$$

where

b = mass in g of the extract (*see* **7.3.4**), and

a = oven-dry mass in g of the test specimen (*see* **7.3.2**).

7.4.1 Determine the mean of all values as obtained in **7.4.**

8 REPORT

8.1 The report shall include the following information:

a) Average moisture content, percent;

b) Average size or finish, percent.

c) Average ash content, percent;

d) Average fatty matter, percent; and

e) Solvent used in the extraction of fatty matter.

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